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(71) Applicant: INSTITUTE DE LA TECHNOLOGIE DU MAGNESIUM, INC. [CA/CA]; 357, rue Franquet, Sainte-Foy, Quebec G1P 4N7 (CA).

(72) Inventors: PEKGÜLERYÜZ, Mihriban, Ozden; 885, du sechoir, Chicoutimi, ~~Quebec G7J 4W9 (CA)~~. ~~LUO~~, Aihua, Alan; Apartment 5, 878, de Bourgogne, Sainte-Foy, Quebec G1X 3E1 (CA).

(74) Agent: BERESKIN & PARR; 40th floor, 40 King Street West, Toronto, Ontario M5H 3Y2 (CA).

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הספריה

(54) Title: CREEP RESISTANT MAGNESIUM ALLOYS FOR DIE CASTING

(57) Abstract

A magnesium based alloy contains from about 2 to about 6 wt.% aluminum and from about 0.1 to about 0.8 wt.% calcium, the balance comprising magnesium. The alloy includes the intermetallic compound Al_2Ca at the grain boundaries of the magnesium crystals. The all y according to this invention may have a creep extension of less than about 0.5 % at about 150 °C.

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Title: CREEP RESISTANT MAGNESIUM ALLOYS FOR DIE CASTING

FIELD OF THE INVENTION

This invention relates to magnesium based alloys. In particular, the invention relates to magnesium based alloys containing aluminum and calcium. The alloys of this invention are particularly useful in die casting applications.

BACKGROUND TO THE INVENTION

Magnesium is the lightest of the structural metals and may readily be fabricated by standard processes. Various magnesium alloys have been developed for use in various applications including, for example, the die casting of parts for automobiles. An example of a magnesium alloy which may be used in the fabrication of automobile parts is AZ91. This magnesium alloy contains about 8.5% aluminum and trace amount of other elements. AZ91 is an economically priced alloy for various applications including the fabrication of automobile parts. One disadvantage of this alloy is that it has a creep extension of about 2.5% (at 150 °C) as measured by ASTM Test No. E139-83. Due to its high creep extension, AZ91 is unattractive in various applications such as components for automobile transmissions where fabricated parts must fit together with high tolerances and remain dimensionally stable during the operating life of the part.

A second magnesium based alloy which is available for use is designated AE42. This alloy comprises about 3.8% aluminum and about 2.4% rare earths together with trace amounts of other elements. This product has a desirable creep extension (about 0.3% or less at 150 °C). Accordingly, while this alloy may be used to fabricate parts having a high degree of dimensional stability. However, due to the use of rare earth elements in fabricating this alloy, the alloy is uneconomical.

SUMMARY OF THE INVENTION

In accordance with the instant invention, there is provided a magnesium based alloy comprising from about 2 to about 6 wt. % aluminum and from about 0.1 to about 0.8 wt. % calcium, the alloy having a creep extension less than about 0.5 %.

Preferably, the amount of aluminum varies from about 2 to about 5 wt. % and, more preferably from about 4 to about 5 wt. %. In addition, the amount of calcium present in the alloy preferably varies from about 0.4 to about 0.8 wt. % calcium and, more preferably, from about 0.5 to about 0.7 wt. % calcium.

As is appreciated from the forgoing, the main constituent elements of the alloy are magnesium, aluminum and calcium. The alloy may also contain other elements (e.g. up to about 0.05 wt. % silicon, up to about 0.2 wt. % zinc, up to about 0.25 wt. % manganese) and impurities (e.g. less than about 0.004 wt. % iron, less than about 0.008 wt. % copper and less than about 0.002 wt. % nickel).

It has surprisingly been found that the addition of the specified amount of aluminum and calcium result in the formation of the intermetallic compound Al_2Ca at the grain boundaries of the magnesium. Without being limited by theory, it is believed that the intermetallic compound Al_2Ca results in high metallurgical stability (due to its high melting point) and strengthens the boundaries of the magnesium grains in the alloy.

Preferably, the alloy comprises from about 1 to about 24 volume % of the intermetallic compound Al_2Ca, more preferably from about 12 to about 24 volume % and, most preferably from about 15 to about 20 volume %.

The alloys of this invention are particularly useful as die casting alloys due to their decreased tendencies of hot-cracking and die-

sticking, their high creep extension and their relative low cost. The alloys may be produced by any standard process used in the industry.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other advantages of the instant invention will be more fully and particularly explained in association with the following discussion of the preferred embodiment of the invention and the following drawings in which:

Figure 1 is a enlargement of the a section of a die cast alloy according to the instant invention;

Figure 2 is a second section of a die cast alloy according to the instant invention;

Figure 3 is a third section of a die cast alloy according to the instant invention; and

Figure 4 is a drawing of an industrial die casting part used in Example 2;

DESCRIPTION OF THE PREFERRED EMBODIMENT

The alloy of this invention comprises magnesium, aluminum and calcium. Calcium and aluminum comprise the main constituent elements in the magnesium based alloy. As discussed below, the alloy may also include other elements as additives or impurities.

The magnesium based alloy preferably contains from about 0.1 to about 0.8 wt. % calcium, more preferably, from about 0.4 to about 0.8 wt. % calcium and, most preferably, from about 0.5 to about 0.7 wt. % calcium. The use of more than about 0.8 wt. % calcium adversely affects the die castability of the alloy due to extensive hot-cracking and die sticking. In addition, if the alloy contains more than

about 0.8 wt. % calcium, the corrosion resistance of the alloy tends to decrease.

The alloy preferably contains from about 2 to about 6 wt. % aluminum, more preferably from about 2 to about 5 wt. % aluminum and, most preferably from about 4 to about 5 wt. % aluminum. If the alloy contains less than about 2 wt. % aluminum, then all of the aluminum will tend to be dissolved in the magnesium. If the alloy contains more than about 6 wt. % aluminum, then the aluminum tends to combine with the magnesium to form significant amounts of the intermetallic compound $Mg_{17}Al_{12}$. This intermetallic compound has a low melting point and accordingly has a deleterious affect on the properties of the magnesium based alloy. The balance of the alloy comprises magnesium.

The magnesium alloy may also include lesser amounts of other additives and impurities. For example, up to about 0.5 wt. % manganese, more preferably from about 0.25 to about 0.5 wt. % manganese may be included in the alloy to improve corrosion resistance. Silicon and zinc are typical impurities which are contained in the magnesium which is used to prepare magnesium alloys. The alloy may contain up to about 0.05 wt. % silicon and up to about 0.2 wt. % zinc. Other elements may be present in amounts up to about 0.01 % each.

Iron, copper and nickel have deleterious affects on the corrosion resistance of magnesium alloys. Accordingly, the alloy preferably contains less than about 0.004 wt. % iron, and more preferably less than about 0.003 wt. % iron, preferably less than 0.008 wt. % copper and preferably less than about 0.002 wt. % nickel and, more preferably, less than about 0.001 wt. % nickel.

It has surprisingly been found that the addition of calcium and aluminum in the weight percents set out herein results in the production of the intermetallic compound Al_2Ca. This intermetallic compound is generally positioned along the grain

boundaries of the magnesium crystals in the alloy. Preferably, the intermetallic continuously surrounds the grains of magnesium. Figures 1 - 3 show micrographs of three different alloys prepared according to the instant invention. The alloy in Figure 1, designated AC50.6 is an alloy according to the instant invention containing 4.5% aluminum and 0.6% calcium. The alloy of Figure 2 is designated AC50.8 and contains 4.5% aluminum and 0.8% calcium. The alloy of Figure 3 is designated AC51 and contains 4.5% aluminum and 1.0% calcium. These micrographs demonstrate the positioning of the intermetallic substantially along the grain boundaries of the magnesium crystals.

The magnesium based alloy of the instant invention has good creep resistance at temperatures commonly encountered by components for cars (e.g. temperatures up to about 150°C). The alloy preferably has a creep extension less than about 2.5%, more preferably less than about 0.5% and, most preferably less than about 0.35%. It will be appreciated that the greater the amount of Al_2Ca which is formed, the lower the creep extension of the alloy. Preferably, the alloy contains from about 1 to about 24 volume % of intermetallic Al_2Ca, more preferably from about 12 to about 24 volume % and, most preferably, from about 15 to about 20 volume %.

The alloy is particularly well adapted for use as a die casting alloy and may be made by any standard die casting process. For example, the alloy may be prepared by charging the constituent elements to a suitable furnace and elevating the constituent elements to a temperature above their melting point. The mixture may be mixed as is known in the art and then poured into a suitable die and cooled to produced the die cast element. Alternately, a magnesium aluminum alloy such as AM50 may be charged to a furnace. Subsequently, after the magnesium aluminum alloy has been melted or substantially melted, calcium, such as in the form of 70/30 Mg-Ca master alloy, may be charged to the furnace. Once this charge has been

melted and mixed, the charge may be poured into a suitable die and cooled to produce the die cast element.

The alloys according to the instant invention have good tensile strength at temperatures up to about 150°C, as measured by ASTM Test No. E8M-90A, and yield strength, as measured by ASTM Test No. E8M-90A. The alloys of the instant invention preferably have a tensile strength greater than about 21 ksi, more preferably greater than about 22 ksi and, most preferably, greater than about 23 ksi. The yield strength is preferably greater than about 12 ksi, more preferably greater than about 13 ksi and, most preferably greater than about 15 ksi.

The invention will be further understood by the following examples which are not to be construed as a limitation of the invention. Those skilled in the art will appreciate that other and further embodiments are obvious and within the spirit and scope of this invention from the teachings of the present examples taken with the accompanying specifications.

Example 1

Several alloys were produced from a magnesium aluminum alloy (AM50) and a calcium magnesium alloy (Mg-Ca master alloy). The composition of these alloys are set out in Table 1:

TABLE 1- COMPOSITION OF STARTING ALLOYS

MATERIAL	Al Wt. %	Ca Wt. %	Cu ppm	Fe ppm	Mn Wt. %	Ni ppm	Si ppm	Zn ppm
AM50 (1)	5.0	0.0002	10	20	0.32	10	70	200
30/70 Mg-Ca (1) MASTER ALLOY	—	30.2	0.08%	0.01%	0.01%	0.001%	0.015%	0.006%

(1) balance is magnesium

These alloys were charged to a 250 tonne frech hot-chamber die casting machine. The feed alloys were raised to a temperature in the range 620°C to about 635°C and mixed. This liquid mixture was then charged into a die to form a test specimen. When

cooled, the die was removed and the test specimens were subjected to tests. The amounts of alloy AM50 and Mg-Ca Master Alloy used in each run, and the composition of the resulting alloys are set out in Table No. 2.

TABLE 2 - COMPOSITION OF DIE CAST ALLOYS

ALLOY	AM50 kg.	Ca-Mg Master Alloy kg.	Al Wt. %	Ca Wt. %	Cu ppm	Fe ppm	Mn Wt. %	Ni ppm	Si ppm	Zn ppm
AC50.6	180	4.0	4.53	0.61	2	10	0.24	1	16	31
AC50.8	180	5.6	4.53	0.85	2	13	0.25	1	20	29
AC51	180	6.7	4.56	1.02	2	20	0.24	2	18	30

Various properties of the alloys where then tested and compared against other magnesium based alloys, namely AZ91 and AE42. The creep resistance of the alloys, was measured according to ASTM Test No. E139-83. The results of the 150°C creep performance tests are set out in Table No. 3.

TABLE 3 - CREEP PERFORMANCE

	AZ91	AE42	AC50.6	AC50.8	AC51
STRESS (MPa)	35	35	35	35	35
TEMPERATURE (°C)	150	150	150	150	150
DURATION (HOURS)	200	200	200	200	200
RUN 1	3.25%	0.257%	0.29%	0.274%	0.287%
RUN 2	2.32%	0.355%	0.346%	0.311%	0.323%
RUN 3	2.04%	0.377%	0.287%	0.208%	0.396%
AVERAGE	2.54%	0.33%	0.31%	0.26%	0.33%

The results demonstrate that the alloys prepared according to the instant invention, namely AC50.6 AC50.8 and AC51 have a creep extension comparable to those of AE42 at 150°C. With a creep extension of only about 0.3%, only slight deformation of structural elements prepared using these alloys will occur over time. The creep extension was one order of magnitude less than the creep extension of standard magnesium based alloy AZ91.

The tensile properties of each of these alloys were also evaluated at 150°C according to ASTM Test No. E8M-90. The results are set out in Table No. 4.

TABLE 4 - TENSILE PROPERTIES

PROPERTY*	AC50.6	AC50.8	AC51	AE42	AZ91
YIELD STRENGTH psi(MPa)	13,754 (95)	14,844 (102)	16,266 (112)	15,513 (107)	15,965 (110)
TENSILE STRENGTH psi(MPa)	22,686 (156)	23,369 (161)	23,973 (165)	23,234 (160)	23,197 (159)
ELONGATION %	8.4	7.4	8.4	36	6.7

*average of three specimens

Table 4 shows that the 150°C tensile strength and yield strength of the alloys prepared according to the instant invention are equal to or better than those of magnesium based alloy AE42. However, the elongation is substantially lower than the elongation of magnesium based alloy AE42.

The salt spray corrosion performance of each of these alloys was then measured pursuant to ASTM Test No. B117. The results are set out in Table No. 5.

TABLE 5 - SALT SPRAY CORROSION

RUN NO.	AE42 mg/cm2/day	AZ91 mg/cm2/day	AC50.6 mg/cm2/day	AC50.8 mg/cm2/day	AC51 mg/cm2/day
Run No. 1	0.1717	0.2362	0.1097	0.1614	0.2317
Run No. 2	0.2509	0.1131	0.1297	0.1817	0.2531
Run No. 3	0.1440	0.1005	0.1265	0.1711	0.2580
Run No. 4	0.1594	0.0777	0.0945	0.1622	0.2220
Run No. 5	0.2888	0.0460	0.1257	0.1780	0.2125
Run No. 6	0.2322	0.0863	0.097	0.1314	0.2245
Average	0.21	0.11	0.11	0.16	0.23

As can be seen from the forgoing table, the alloys of the instant invention have similar corrosion resistance to magnesium based alloy AZ91 and AE42. In fact, alloy AC50.6, which contains only about 0.6% calcium, has a corrosion resistance which is twice that of alloy AE42. Accordingly, by selecting an alloy according to the instant invention having a lower level of calcium, the corrosion resistance of the resulting alloy may be substantially improved.

Example 2

Two alloys with different calcium levels were prepared by the procedure of Example 1 and cast into an industrial die for die castability study.

The alloys were charged to a 600 tonne Prince cold-chamber die casting machine. The feed alloys were raised to a temperature of 660°C and mixed. This liquid mixture was then charged into an industrial die having five zones numbered 1 - 5 through a siphon tube to form an industrial die casting as shown in Figure 4. When cooled, the casting was removed from the die. One hundred (100) castings of each alloy were produced and subjected to die castability evaluation and mechanical testing. Both visual and real-time X-ray inspections were used to detect the casting defects such as cracks and incomplete filling. Test specimens for tensile and creep testing were then machined from zone 3 of the die castings as shown in Figure 4.

The amounts of alloy AM50 and Mg-Ca Master Alloy used in each run, and the composition of the resulting alloys are set out in Table No. 6.

TABLE 6 - COMPOSITION OF DIE CAST ALLOYS

Alloy	AM50 kg	Mg-Ca Master Alloy kg	Al Wt. %	Ca Wt. %	Cu ppm	Fe ppm	Mn Wt. %	Ni ppm	Si ppm	Zn ppm
AC50.7	780	18.6	4.40	0.68	4	16	0.26	2	10	200
AC51	780	29.0	4.40	0.99	4	18	0.25	2	10	200

The die castability evaluation results obtained from the visual real-time X-ray inspections demonstrate that the alloy AC50.7 is readily die castable with very few casting defects. However, in the production of die castings using alloy AC51, substantial die-sticking problems occurred, and significantly more casting defects such as cold shots, cracks and sink marks were observed in the castings.

The results show that the use of more than about 0.8 wt. % calcium adversely affects the die castability due to the increased tendencies of hot-cracking and die-sticking during die casting productions. The magnesium based alloys of this invention preferably contain up to 0.7 wt. % of calcium.

The creep and tensile properties of the alloys were tested and compared against other magnesium based alloys, namely AZ91 and AM50. The results of the 150°C creep performance tests are set out in Table No. 7.

TABLE NO. 7 - CREEP PERFORMANCE

	AZ91	AM50	AC50.7	AC51
Stress (MPa)	35	35	35	35
Temperature (°C)	150	150	150	150
Duration (Hours)	200	200	200	200
RUN 1	2.34%	1.58%	0.159%	0.145%
RUN 2	1.88%	1.67%	0.146%	0.145%
RUN 3	2.34%	3.21%	0.218%	0.217%
AVERAGE	2.19%	2.15%	0.17%	0.17%

The results show that the creep extension of the alloys prepared according to the instant invention is about one order of magnitude less than those of standard magnesium based alloys AZ91 and AM50. The results also demonstrate that the creep resistance of the alloys of this invention can be obtained with a calcium content as low as 0.6 to 0.7 wt. %.

The tensile properties of these alloys were also tested at 150°C, and the results are set out in Table No. 8.

TABLE 8 - TENSILE PROPERTIES

PROPERTY*	AC50.7	AC51	AZ91	AM50
YIELD STRENGTH psi (MPa)	15,100 (104.1)	16,700 (115.4)	15,100 (104.3)	11,700 (80.4)
TENSILE STRENGTH psi(MPa)	25,900 (178.8)	26,000 (179.3)	27,800 (191.9)	23,200 (159.9)
ELONGATION %	16.7	18.3	13.3	24.0

* average of three specimens

Table 8 shows that the 150°C tensile strength and yield strength of the alloys prepared according to the instant invention are equal to or better than those of magnesium based alloy AZ91 or AE42. The elongation is higher than that of AZ91, but substantially lower than that of AM50.

WE CLAIM:

1. A magnesium based alloy comprising magnesium, from about 2 to about 6 wt. % aluminum and from about 0.1 to about 0.8 wt. % calcium, said magnesium forming magnesium crystals in said alloy, at least a portion of said aluminum and calcium forming the intermetallic compound Al_2Ca, said Al_2Ca being located at the grain boundaries of said magnesium crystals.

2. The alloy as claimed in claim 1 wherein said intermetallic compound comprises from about 1 to about 24 volume % of said alloy.

3. The alloy as claimed in claim 1 wherein said intermetallic compound comprises from about 12 to about 24 volume % of said alloy.

4. The alloy as claimed in claim 1 wherein the main constituent elements are magnesium, aluminum and calcium.

5. The alloy as claimed in claim 2 wherein said alloy has a creep extension less than about 0.5% at about 150°C.

6. A magnesium based alloy comprising from about 2 to about 6 wt. % aluminum and from about 0.1 to about 0.8 wt. % calcium, said alloy having a creep extension less than about 0.5 % at about 150°C.

7. The alloy as claimed in claim 6 wherein said alloy comprises from about 2 to about 5 wt. % aluminum and from about 0.4 to about 0.8 wt. % calcium.

8. The alloy as claimed in claim 6 wherein said alloy comprises from about 4 to about 5 wt. % aluminum and from about 0.5 to about 0.7 wt. % calcium.

9. The alloy as claimed in claim 6 wherein said alloy further comprises less than about 0.05 wt % silicon, less than about 0.2 wt. % zinc, less than about 0.5 wt. % manganese, less than about 0.0004 wt % iron, less than about 0.008 wt. % copper and less than about 0.002 wt. % nickel.

10. The alloy as claimed in claim 6 wherein said alloy is a die casting alloy.

11. A process for producing cast magnesium based alloy articles having decreased elongation comprising the steps of:

a. forming a molten alloy comprising from about 2 to about 6 wt. % aluminum and from about 0.1 to about 0.8 wt. % calcium, the remainder essentially being magnesium; and,

b. molding said molten alloy and solidifying to form the cast alloy article,

said magnesium forming magnesium crystals in said alloy, at least a portion of said aluminum and calcium forming the intermetallic compound Al_2Ca, said Al_2Ca being located at the grain boundaries of said magnesium crystals..

12. The process as claimed in claim 11 wherein said intermetallic compound comprises from about 1 to about 24 volume % of said alloy.

13. The process as claimed in claim 11 wherein said intermetallic compound comprises from about 12 to about 24 volume % of said alloy.

14. The process as claimed in claim 12 wherein said alloy comprises from about 2 to about 5 wt. % aluminum and from about 0.4 to about 0.8 wt. % calcium.

15. The process as claimed in claim 12 wherein said alloy comprises from about 4 to about 5 wt. % aluminum and from about 0.5 to about 0.7 wt. % calcium.

16. The process as claimed in claim 12 wherein said alloy has a creep extension less than about 0.5%.

17. A process for producing cast magnesium based alloy articles having a creep extension less than about 0.5 % at about 150°C comprising the steps of:

a. forming a molten alloy comprising from about 2 to about 6 wt. % aluminum and from about 0.1 to about 0.8 wt. % calcium, the remainder essentially being magnesium; and,

b. molding said molten alloy and solidifying to form the cast alloy article.

18. The process as claimed in claim 17 wherein said alloy comprises from about 2 to about 5 wt. % aluminum from about 0.4 to about 0.8 wt. % calcium.

19. The process as claimed in claim 17 wherein said alloy comprises from about 4 to about 5 wt. % aluminum from about 0.5 to about 0.7 wt. % calcium.

20. The process as claimed in claim 17 wherein said alloy further comprises less than about 0.05 wt % silicon, less than about 0.2 wt. % zinc, less than about 0.5 wt. % manganese, less than about 0.0004 wt % iron, less than about 0.008 wt. % copper and less than about 0.002 wt. % nickel.

FIGURE 1

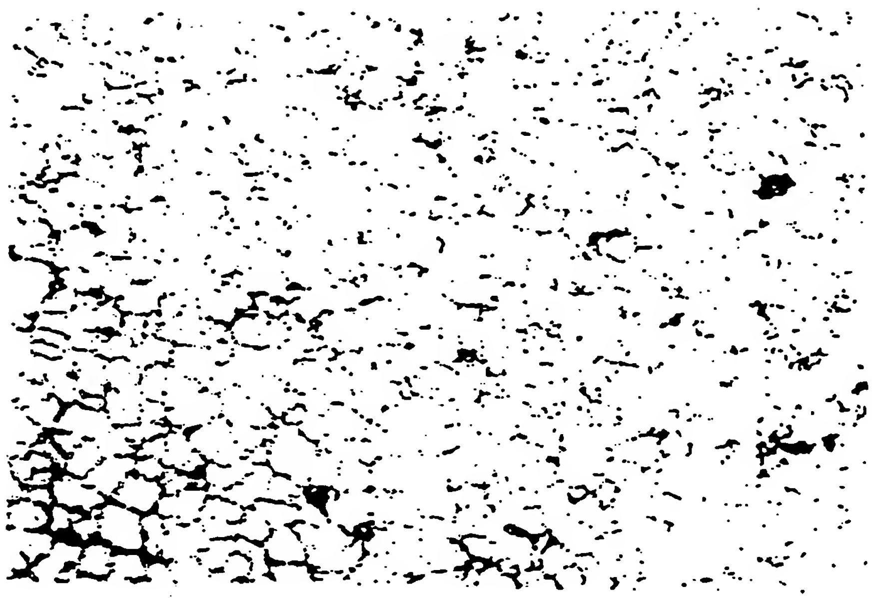

FIGURE 2

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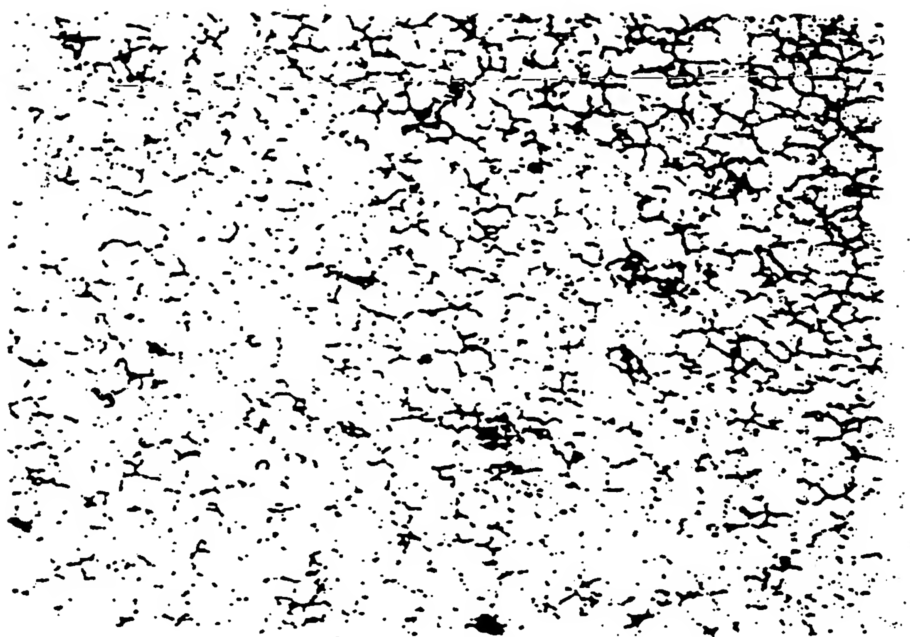
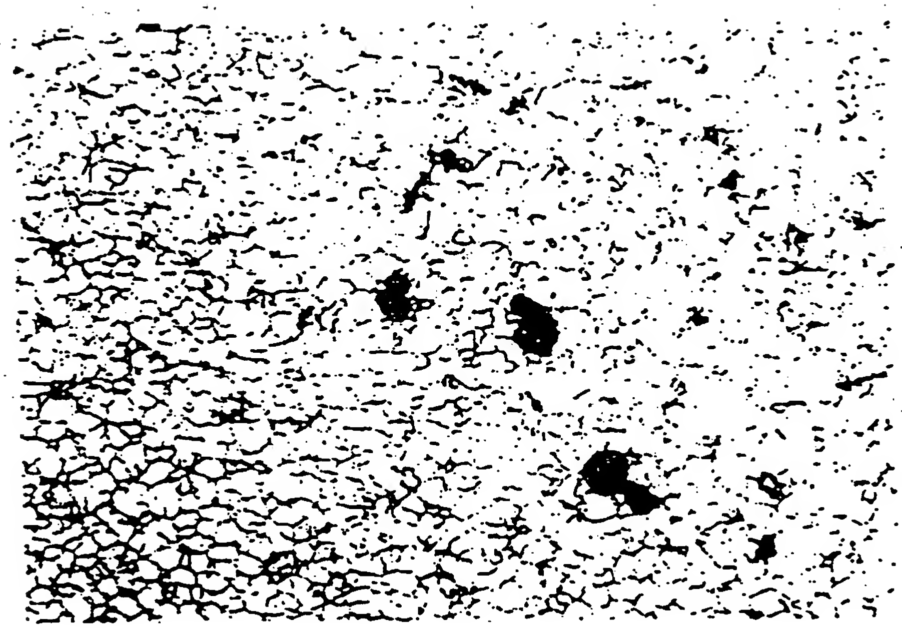


FIGURE 3



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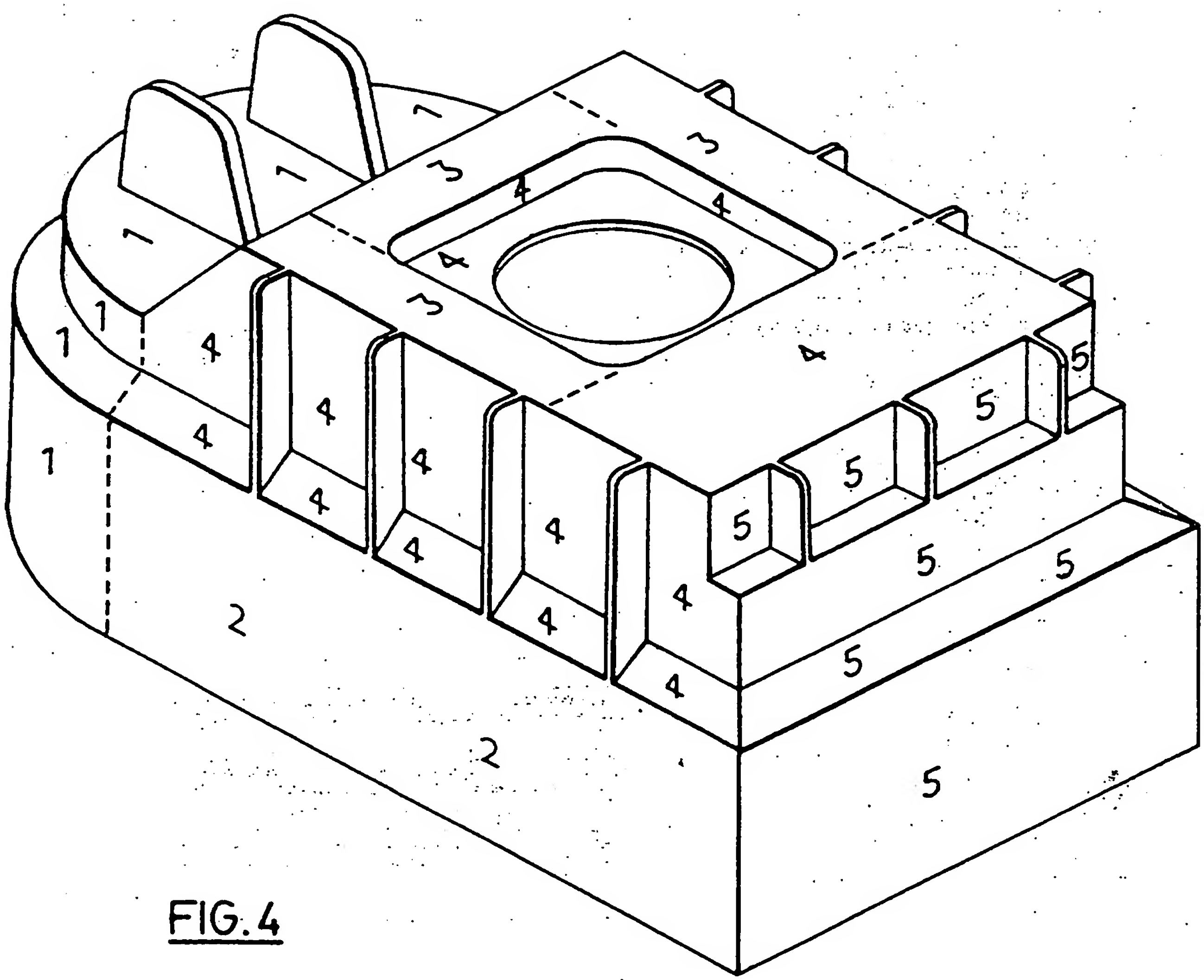

FIG. 4

INTERNATIONAL SEARCH REPORT

Inter nal Application No
PCT/CA 96/00091

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C22C23/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JOURNAL OF NUCLEAR MATERIALS, vol. 82, no. 1, 1979, pages 39-53, XP000573642 E.F. STURCKEN: "Irradiation Effects in Magnesium and Aluminium Alloys" p. 47, right hand col. last paragraph - p. 50, left hand col, paragraph 2 see page 40, column 2, paragraph 2.1 - page 41; figures 7,8; table 1 ---	1-6, 9-13,16, 17,20
Y	DE,A,25 26 024 (MAHLE GMBH) 15 July 1976 cf. the whole document see claims 1,2 ---	6-8,14, 15,18,19
Y	WO,A,89 08154 (PECHINEY ELECTROMETALLURGIE) 8 September 1989 see claims 1,3; table 1 -----	6-8,14, 15,18,19

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European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Ries, R

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